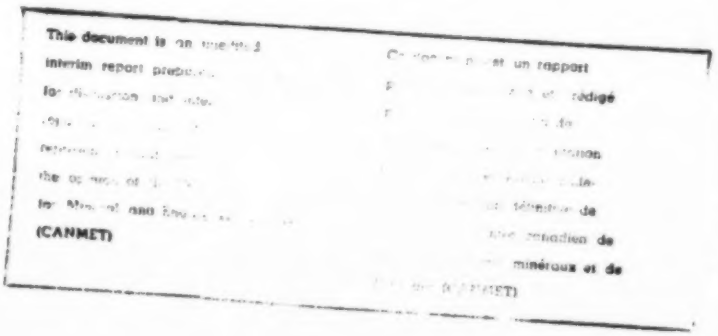
Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

CANMET
Canada Centre for Mineral and Energy Technology
Centre canadien de la technologie des minéraux et de l'énergie

THE URANIUM ANALYTICAL LABORATORY

J.L. Dalton
Chemical Laboratory

December 1986


This document is an unedited
interim report prepared
for publication but rather
reference in
the opinion of
for Mineral and Energy
(CANMET)

rédigé
de
minéraux et de

Project: 30.69.02
Analytical Methodology

13 pp

MINERAL SCIENCES LABORATORIES
DIVISION REPORT MSL 86-167 (TR)

MICROMEDIA

THE URANIUM ANALYTICAL LABORATORY

by

J.L. Dalton

ABSTRACT

The Division of Nuclear Fuel Cycle of the International Atomic Energy Agency requested the assistance of CANMET in the preparation of a manual on the analytical chemistry in the uranium industry. The staff of the Chemical Laboratory has responded to this request and this report is intended to be part of the contribution to the analytical chemistry manual. This report deals with the management aspects of operating an analytical laboratory for the uranium industry.

Key Words: Analysis, Management, Calibration, Quality Control

---

* Section Head, XRF and Radioanalysis Section, Chemical Laboratory, Mineral Sciences Laboratories.

LABORATOIRE D'ANALYSE DE L'URANIUM

par

J.L. Dalton

RÉSUMÉ

La Division du cycle du combustible nucléaire de l'Agence internationale de l'énergie atomique a demandé au CANMET de participer à la préparation d'un manuel sur la chimie analytique pour l'industrie de l'uranium. Le personnel du Laboratoire de chimie a répondu à cette demande et le présent rapport reflète une partie de sa contribution au manuel sur la chimie analytique. Ce rapport a donc pour objet la gestion d'un laboratoire d'analyse au niveau de son fonctionnement dans le cadre de l'industrie de l'uranium.

THE URANIUM ANALYTICAL LABORATORY

The purpose of this chapter is to highlight the management aspects of operating an analytical laboratory. The assumptions made are that the laboratory is located in an underdeveloped country and will be the central analytical laboratory for the uranium industry in that country. Thus, the laboratory will have two functions; the analysis of samples arising from geological survey parties and also a a mining/milling operation.

The major responsibilities of the Laboratory are as follows:

(i) Analysis of field samples from exploration projects. There may be a great number of samples from these projects, most of which will require some form of sample preparation prior to analysis. Extensive sample storage facilities will also be a requirement for this project.

(ii) Analysis of solid samples from the mining operation, which will require sample preparation and rapid turn-around time.

(iii) Analysis of mill process samples such as powdered head samples and leach residues, aqueous and organic solutions and concentrates. With the exception of the concentrates, these samples will require rapid turn-around time.

(iv) Analysis of uranium ore concentrates, which require a high degree of accuracy in the determination of uranium.

(v) Analysis of environmental monitoring samples.

(vi) Analysis of occupational health and safety samples, such as urine.

(vii) Issuance of appropriate analytical reports and the maintenance of a report record-system.

(viii) The maintenance of a sample preparation and sample storage facility.

In order to accomplish the above, the Laboratory will have several operating units as shown graphically on the organisation chart. Senior staff consists of a Chief Chemist and Deputy Chief Chemist, supervisory staff is composed of working level graduate chemists and/or chemical technologists and working staff consists of about 20-30 people who have had some formal education in chemistry and preferably analytical chemistry.

A convenient management method is to divide the work by type of project with several sections or operating units dealing with essentially different types of samples, as shown in the organisation chart, Fig. 1. A good reason for this division is to prevent sample cross-contamination which could occur if the same worker deals with both ore concentrates and exploration samples with the same facilities during a shift. This division should also be physical, with different locations within the laboratory. It is suggested that there be about five graduate chemists or the equivalent in the organisation in supervisory capacities as shown on the organisation chart. The above recommendations are based on an assumption that there is enough analytical work to support the four main operating units. If that is not the case, then overlap will be necessary, with less staff required than as shown on the organisation chart.

The ready availability of instrumentation service is an operational requirement of the analytical control laboratory to effect rapid emergency repairs to instrumentation that is used in critical functions. Control laboratories cannot afford to wait lengthy periods of time to have instrumentation repaired at the place of the manufacture. If this service is not available through the mine/mill complex, then the Chief Chemist will have to have staff or contract services readily available to meet this requirement. A major responsibility of the stores staff will be to ensure that instrumentation consumables and replacements are readily available. The delivery time for instrumentation parts can often take months and therefore a stock for immediate access is necessary.

Priorities and scheduling of analytical services should be mutually agreed upon before the laboratory commences operation. It is very important that the mine site objectives and goals are understood by the managment of the Analytical laboratory. There should also be periodic reviews of policy because goals may change with time. Implementation of established priorities and schedules will largely be a responsibility of the Deputy Chief Chemist. There could very well be an operational conflict between the analysis of a large number of field samples, which require some form of sample preparation and the relatively small number of samples originating from the mine/mill control function. In order to resolve this potential conflict, it may be necessary to operate two separate functions in the

Laboratory, including expensive analytical instrumentation, in order to satisfy the objectives of each of these two functions.

The Chief Chemist and the Deputy Chief Chemist should be university graduates in chemistry and possess a strong background in analytical chemistry with several years of experience in the uranium industry. Both positions require good organizational and communication skills, self-motivation and the ability to work with people. The Chief Chemist should have received management training.

There will be several people reporting to the Chief Chemist, such as the Deputy Chief Chemist, the office staff, storesperson/laboratory steward and the head of the environmental monitoring group. The office staff will have to deal with correspondence, internal management documents such as purchase requisitions and payroll data, issuing of official reports and the maintenance of an analytical records system. The responsibilities of the stores/laboratory steward will be to ensure the ready availability of chemicals, laboratory equipment and glassware, assistance given in sample recording and distribution and may also have some responsibility in maintaining cleanliness in the laboratory operations, depending upon the workload of any one of the preceeding three items.

It is anticipated that the uranium mining/milling operation will be subject to environmental constraints, such as regulations governing the escape of pollutants from the site and the health and safety of the whole site workforce. The monitoring of effluents from the tailings ponds, which should include both collection and analysis of suitable samples should be controlled by an experienced chemist, reporting to the Chief Chemist. The head of this Section will also be involved in Health and Safety matters, such as the analysis of urine for uranium.

The staff in charge of the four working groups will report to the Deputy Chief Chemist, who will be responsible for arranging the analytical work of the laboratory. As mentioned previously, there may be difficulties in having analytical work from both exploration parties and mine/mill control carried out in a central facility, if common instrumentation is shared between both functions. The Deputy Chief Chemist will have to schedule the work so that the objectives of both functions are met, and any changes to schedules and priorities should only be carried out by this person. Another responsibility will be the rotation of staff from the

technical pool among the various groups. These assignments should be made every 6-12 months. It is recommended that all staff, including professionals, rotate periodically so that resource personnel are available for all functions of the laboratory. The Deputy will also be responsible for laboratory quality control.

The Management Information System (MIS) will be an important component of the laboratory. The MIS can serve a variety of uses such as recording sample details and issuing sample numbers, documenting analytical progress and results, and the printing of analytical and management reports. The MIS can also handle normal office routines such as purchasing records, personnel data, and shift scheduling. Maintenance of analytical records is also an important part of the MIS operation. Analytical records should be maintained for at least two years.

The MIS will have an important role in the quality control operation of the laboratory. The MIS software should be designed so that the retrieval of actual sample data is accompanied with quality control data. Quality control data should be filed with the particular sample data and absence of quality control data should be highlighted.

Operating the MIS will be carried out by several people, none of whom will be assigned to the system on a full time basis. Office personnel, laboratory steward(s) and the supervisory analytical staff will all have to be familiar with the system, albeit some more than others. The Deputy Chief Chemist and some of the supervisory staff should be very familiar with the system. Turn-key MIS systems can be purchased but most operations have sufficient unique features that the services of a programmer/analyst will be necessary at the time of implementing the system. It is extremely important to have good documentation on the system prior to acceptance. It is suggested that there be several terminals conveniently located throughout the work site such as the Laboratory office, the laboratory itself, mill and mine control rooms. In general, a terminal should be located wherever samples originate. However, only certain commands should be available on client's terminals. These commands should be restricted to status and report functions. No system imput, other than queries, should be allowed from the client's terminals.

A clerical records system will have to be designed and implemented if a computer-based MIS system is not available. A clerical system will probably require more permanent staff than the computer system. The clerical staff

will have to maintain the sample entry system, manually issuing laboratory numbers and recording such items as sample identification, level of priority, analytical work requested, a system to report the analytical results and the compilation of management documents such as daily, weekly, monthly and yearly reports. Paper-work can be expedited a great deal if well-designed forms and/or laboratory worksheets are available.

Quality control will be a very important on-going feature of the analytical laboratory's work. Policy with respect to Q.C. on various analytical tasks should be worked out to a mutual satisfaction between laboratory management and client. Aspects that have to be settled are the turn-around times and analytical tolerances. For instance, in exploration programmes or mill control it may be much more important to have rapid turn-around time with ± 10% relative error than slow turn-around time with ± 3% relative error. Uranium ore concentrate, on the other hand, requires a very precise and accurate analysis because these provide the basis for financial return to the company.

Accuracy of analysis should be established for the various analytical tasks by use of certified reference materials and by primary standards, or by participating in interlaboratory programmes on various representative samples from test work. Once representative samples have been analysed and it is known that the samples will not deteriorate with time, then they can be used in a continuing Q.C. program.

Q.C. programs should be in use to monitor all the laboratory's analytical work. Depending upon the critical nature of the analytical work, quality control should vary between 5% and 20% of the work load. In order to meet environmental considerations, it may be necessary to have a standard with every four solutions for Ra-226 determinations. For uranium in exploration samples, 5 standards per 100 samples may be quite sufficient. The amount of Q.C. is dependent upon the use of the data and the acceptable control limits established for that particular determination. Quality control policy is very much unique to a particular location and varies a great deal from one operation to another, even within the same industry. It must be emphasized that Q.C. is an integral part of the laboratory's operation and cannot be overlooked. Quality control assumes a very important role if working staff is constantly changing.

Health and safety aspects of the laboratory operation should be established and supervised by the Chief Chemist, as this person is ultimately responsible for the safe conduct of work and elimination of hazardous practices in the laboratory. There are several factors that should be considered such as the proper operation of fume hoods with due regard to the safe use of perchloric acid, the proper storage of chemicals, the ready availability of personal safeguards and installation of emergency shower and eye-wash fountains and access to radiation monitoring equipment. Health and safety matters should be periodically reviewed by the appropriate laboratory staff. As much of the mill process work is of a chemical nature, the Chief Chemist may also be asked to participate in the establishment and review of health and safety practices in the mill, for instance, the analysis of urine for uranium for the mill staff.

A possible design for an analytical chemical facility is shown in Fig. 2. The facility is large enough to accommodate about 30 people with a capacity of tens of thousands determinations per month assuming that the laboratory is equipped with modern instrumentation, and the nature of a substantial portion of the work is similar. The dimensions given in Fig. 2 are intended as guidelines.

The acid hoods should have adequate ventilation, hot plates, electrical, gas, purified and tap water service and lighting. It is recommended that all hoods be capable of handling perchloric acid with washdown facilities.

Each work bench should have adequate electrical, gas and water services. It is also convenient to have some pressurized air and vacuum outlets in the laboratory. The workbenches shown in Fig. 2 are some 7 metres long to accommodate 2 workers per aisle. Ideally, each worker should have access to about 3 metres of double bench space. The benches should be equipped with shelves and drawers for storage of glassware, labware and sundry devices. The plan shown in Fig. 2 should be able to accommodate 10 bench workers in the central area.

Because of the possibility of contamination between samples, the work load is physically divided into three distinct areas. Both exploration and mine/mill control have their own sample preparation and work bench areas. Uranium ore concentrate analysis has a special room. Glassware should be restricted to one work area.

Ideally, the whole laboratory should have a constant operating temperature. If that is not possible then the three instrumentation rooms, uranium ore concentrate analysis and radiochemistry will have to have a constant operating temperature. Many instrumentation problems can be avoided by having a year round constant operating temperature. The accepted tolerance for constant operating temperature is ± 2°C. The laboratory should also be fitted with isolation transformers, constant voltage units and filters to maintain operational electrical service.

The instrumentation rooms will have to be fitted with some canopy hoods for the operation of atomic absorption and inductively coupled plasma spectrometers.

Samples arrive in the reception area where the appropriate information is entered into the MIS system, sample numbers are assigned along with possible management information such as level of priority, assignment to appropriate analytical programs and any special considerations. It should be possible to expedite the documentation by having standard forms. Information required would be, for example, sample origin, management code, date and time and the required analytical work indicated by tick marks in appropriate boxes. Once documentation is complete, the sample is forwarded to sample preparation if necessary, to the relevant laboratory supervisor or included in established laboratory practice. The supervisor will decide how and when the analytical work will be carried out, depending upon previously established management criteria. The Deputy Chief Chemist should be notified of the arrival of any unusual analytical work.

The style of reporting the analytical results is a characteristic of the particular management style and client's needs. If the laboratory has a computer-based MIS, then all results are entered into the system and are available on the client's terminals on demand. Hard-copy print-out is available whenever desired. If the laboratory does not have a computer-based system, the results from the mine/mill complex will probably be phoned to the appropriate process control location. The hand-written work-sheets or report summary sheets will be filed in either the laboratory office or with the pertinent supervisor. It is important to have records of all analytical work for possible future reference and also for the preparation of management reports. The survey work, and some of the

mine/mill work, will certainly require the preparation and issuance of an official report. These reports will contain sample number, sample description, a possible methodology reference and the analytical result.

The concept of reporting estimates of accuracy along with the analytical result is a contentious issue. Some analysts claim that it is not necessary for control work when the process and the analytical methodology is known to be under control. In this case, accuracy in the analytical results are generally well-known, and the reporting of accuracy estimates with every result adds costs to the job without an apparent benefit. For survey work, however, it may very well be necessary to have an estimate of accuracy with some elements. The estimate of accuracy is generally reported as ± figures following the analytical results. If possible, the ± figures are obtained from the analysis of certified reference materials. For solution work, the accuracy estimates may be obtained by statistical analysis of the analytical data. It should be pointed out that replicate analysis of calibration solutions will only provide estimates of precision, not accuracy. NBS Special Publication 300 contains many articles on estimating precision and accuracy from scientific and engineering measurements (1).

REFERENCES

1. "Precision Measurements and Calibration", Special Publication 300 United States Department of Commerce, National Bureau of Standards, Stock No. 003-003-00072-8.

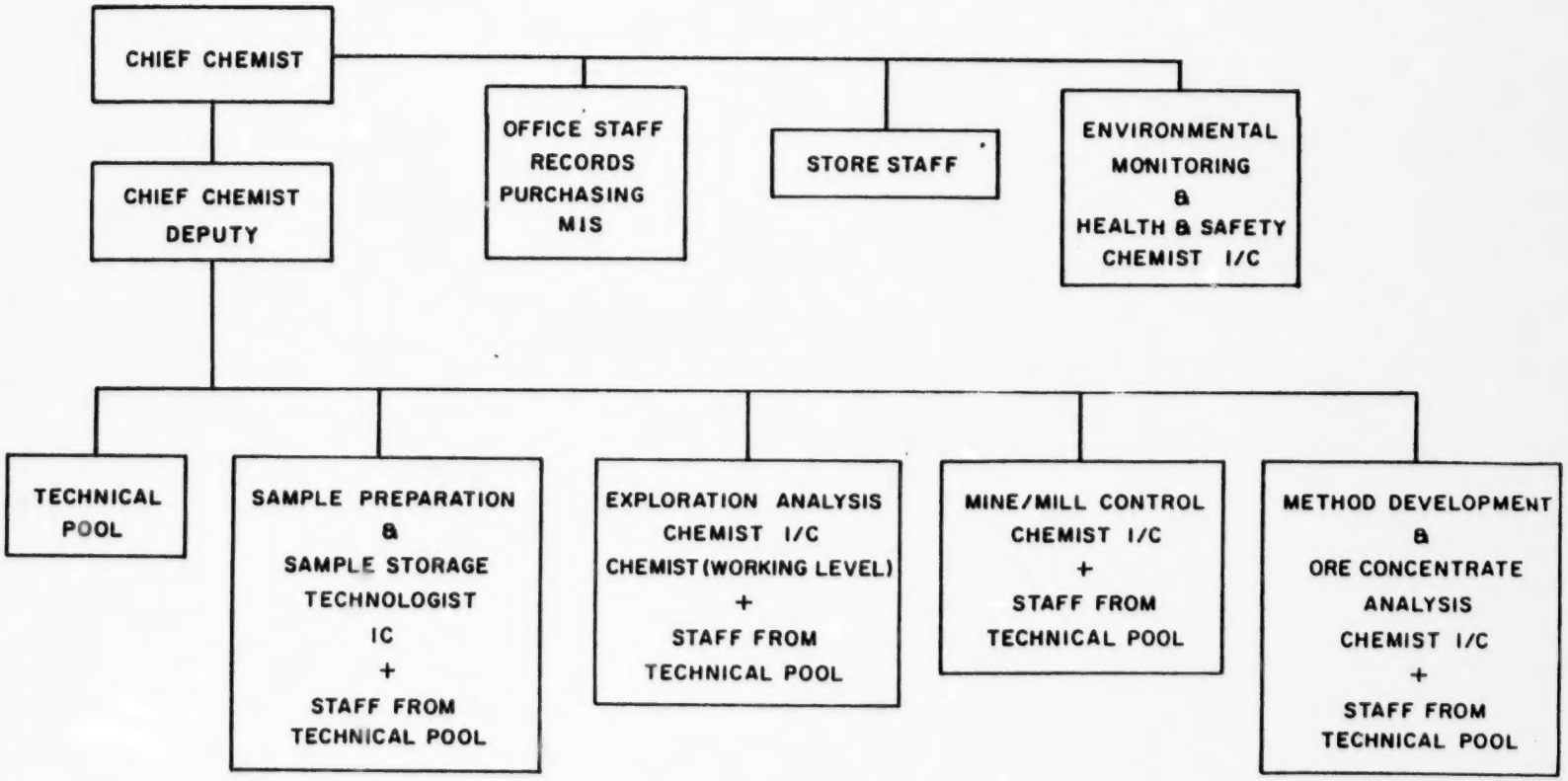

CHIEF CHEMIST
CHIEF CHEMIST DEPUTY
OFFICE STAFF
RECORDS
PURCHASING
MIS
STORE STAFF
ENVIRONMENTAL MONITORING & HEALTH & SAFETY CHEMIST I/C
TECHNICAL POOL
SAMPLE PREPARATION & SAMPLE STORAGE TECHNOLOGIST IC + STAFF FROM TECHNICAL POOL
EXPLORATION ANALYSIS CHEMIST I/C CHEMIST (WORKING LEVEL) + STAFF FROM TECHNICAL POOL
MINE/MILL CONTROL CHEMIST I/C + STAFF FROM TECHNICAL POOL
METHOD DEVELOPMENT & ORE CONCENTRATE ANALYSIS CHEMIST I/C + STAFF FROM TECHNICAL POOL

Figure 1. Organization Chart for the Uranium Analytical Laboratory

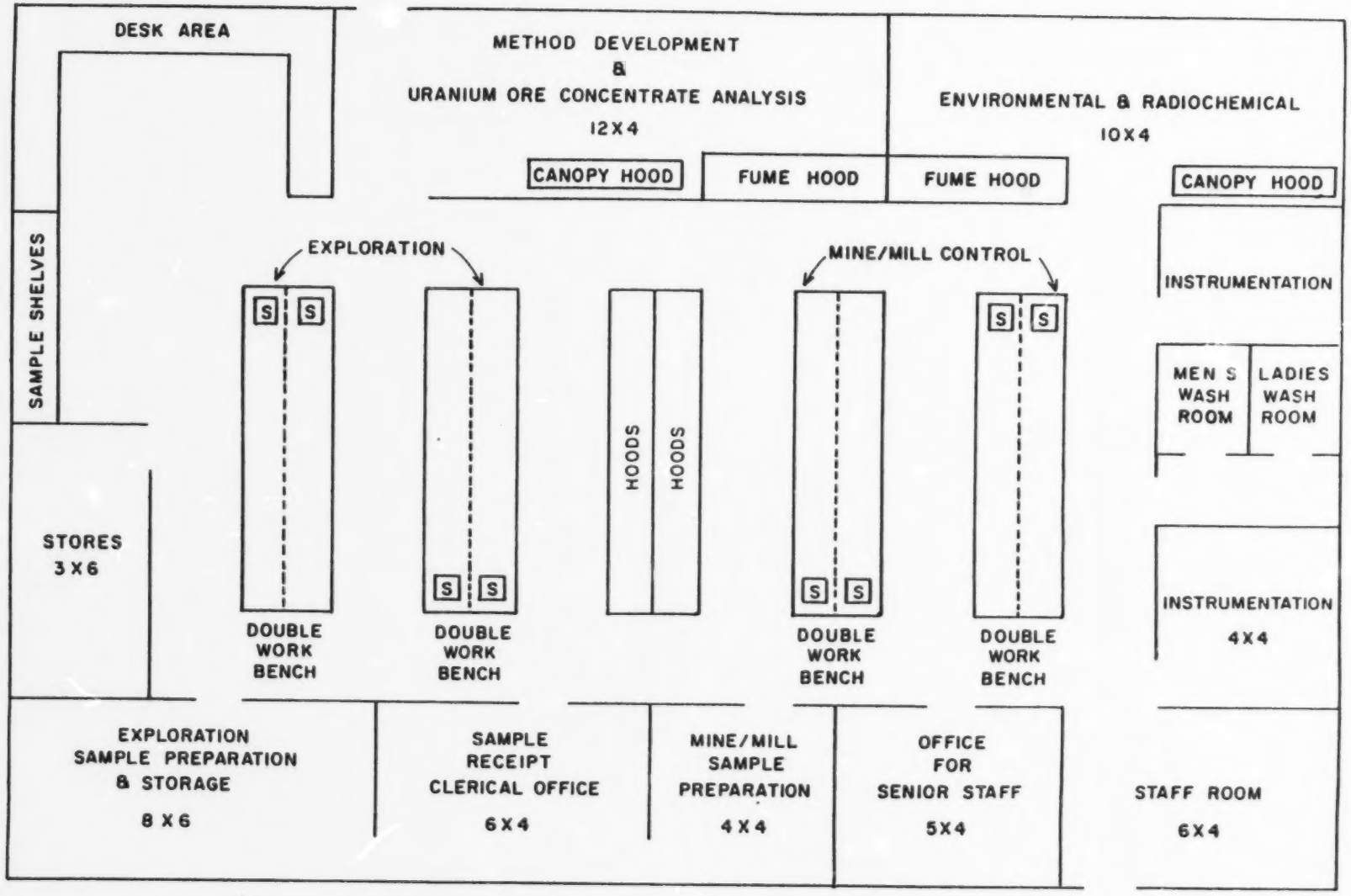


Figure 2 Suggested Layout for the Uranium Analytical Laboratory. (29 X 19 M)